

# 1.0 Introduction

This appendix documents the results of a quality assurance (QA) review of the analytical data for storm water samples collected as part of the Terminal 4 Storm water Project. The data reviewed includes storm water sample data collected during sampling performed on March 24, 2007 through January 26, 2008. The samples were analyzed by Columbia Analytical Services (CAS) of Kelso, Washington, and Vista Analytical Laboratory (Vista) or El Dorado Hills, California.

The QA review outlines the applicable quality control criteria utilized during the data review process, as well as any deviations from those criteria. Examination and validation of the laboratory summary report, includes:

- Analytical methods;
- Reporting limits;
- Detection limits and estimated concentrations;
- Sample holding times;
- Custody records and sample receipt;
- Spikes, blanks, and surrogates;
- Duplicates; and
- Calibration and internal standard.

The QA review did not include a review of raw data. Section 2.0 lists the analytical methods used in sample analysis. Section 3.0 defines the QA terms used in this report. Section 4.0 provides the QA results for each sampling event. Section 5.0 lists the qualifiers used in the tabulated results. A list of abbreviations used in this report is included at the end of the document for reference.

## 2.0 Analytical Methods

Chemical analyses on storm water samples consisted of one or more of the following, unless otherwise noted:

- Total and dissolved organic carbon (TOC and DOC) by EPA Method SW9060/415.1/SM-5310C;
- Total suspended solids (TSS) by EPA Method 160.2;
- Turbidity by EPA Method 180.1;
- Total and dissolved metals by EPA Method 6020;

- Total and dissolved mercury by EPA Method 7470A;
- Total and dissolved total petroleum hydrocarbons (TPH) as oil and grease by EPA Method 1664;
- Total and dissolved phthalates by EPA Method 525.2;
- Total and dissolved organochlorine pesticides by EPA Method 8081;
- Total and dissolved polychlorinated biphenyl (PCB) aroclors by EPA Method 8082;
- Total and dissolved polynuclear aromatic hydrocarbons (PAHs) by EPA Method 8270C; and
- Total and dissolved PCB congeners by EPA Method 1668.

# 3.0 Quality Assurance Objectives and Review Procedures

The general QA objectives for this project were to develop and implement procedures for obtaining, evaluating, and confirming the usability of data of a specified quality for monitoring upland stormwater. To collect such information, analytical data must have an appropriate degree of accuracy and reproducibility, samples collected must be representative of actual field conditions, and samples must be collected and analyzed using unbroken chain-of-custody procedures.

Reporting limits and analytical results were compared to action levels for each parameter in the media of concern. Precision, accuracy, representativeness, completeness, and comparability parameters used to indicate data quality are defined below.

Reporting Limits. Method reporting limits (MRLs) are set by the laboratory and are based on instrumentation abilities, sample matrix, and suggested MRLs by the U.S. Environmental Protection Agency (EPA) or the Department of Environmental Quality (DEQ). In some cases, the MRLs are raised due to high concentrations of analytes in the samples or matrix interferences. MRLs are generally consistent with industry standards and below promulgated regulatory standards when possible (if not raised, as discussed above).

**Detection Limits and Estimated Concentrations.** The method detection limit (MDL) is the lowest quantity of a substance that can be distinguished from the absence of that substance within a stated confidence limit. The MDL is estimated from the mean of the blank, the standard deviation of the blank and some confidence factor. Performing the sample preparation has potential to underestimate the true MDL.

**Holding Times.** Holding times are the length of time a sample can be stored after collection and prior to analysis without significantly affecting the analytical results. Holding times vary with the analyte, sample matrix, and analytical methodology used to quantify the analyte's concentration.

**Custody Records and Sample Receipt.** Chain of custody (COC) refers to the document or paper trail showing the seizure, custody, control, transfer, analysis, and disposition of physical and electronic evidence. The sample receipt identifies the condition of samples upon arrival at the analytical laboratory.

**Method Blanks.** A method, or laboratory, blank is a sample prepared in the laboratory along with the actual samples and analyzed for the same parameters at the same time. It is used to assess if detected contaminants may have been the result of contamination of the samples in the laboratory.

**Laboratory Control Sample**. A laboratory control sample (LCS) is analyzed by the laboratory to assess the accuracy of the analytical equipment. The sample is prepared from an analyte-free matrix that is then spiked with known levels of the constituents of interest (i.e., a standard). The concentrations are measured and the results compared to the known spiked levels. This comparison is expressed as percent recovery.

The LCS analyses for the PCB congeners (completed by Vista) are referred to in the laboratory reports as the on-going precision and recovery standard (OPR). The laboratory project manager verified that the OPR is equivalent to the LCS.

Laboratory Control Sample Duplicate. In addition, a second laboratory control sample (i.e., the laboratory control sample duplicate [LCSD]) is prepared as above and analyzed. This is compared to the initial laboratory control sample to assess the precision of the analytical method (i.e., relative percent difference [RPD]).

Matrix Spike Analyses. Matrix spike (MS) analyses are performed on samples submitted to the laboratory that are of the same matrix as the actual sample. It is spiked with known levels of the constituents of interest. These analyses are used to assess the potential for matrix interference with recovery or detection of the constituents of interest and the accuracy of the determination. The spiked sample results are compared to the expected result (i.e., sample concentration plus spike amount) and reported as percent recovery.

Lab Duplicate. A laboratory duplicate is a second analysis of the QA/QC sample, which serves as an internal check on laboratory quality as well as potential variability of the sample matrix. The laboratory duplicate is analyzed and compared to the primary sample analysis to assess the precision of the analytical method. This comparison can be expressed by the RPD between the original and duplicate sample.

**Surrogate Recovery.** Surrogates are organic compounds that are similar in chemical composition to the analytes of interest and spiked into environmental and batch QC samples prior to sample preparation and analysis. Surrogate recoveries for environmental samples are used to evaluate matrix interference on a sample specific basis. Surrogates are organic compounds that are similar in chemical composition to the analytes of interest and spiked into environmental and batch QC samples prior to sample preparation and

analysis. Surrogate recoveries for environmental samples are used to evaluate matrix interference on a

sample specific basis.

The surrogate recovery analyses for the PCB congeners (completed by Vista) are referred to in the laboratory reports as the Internal Standard. The laboratory project manager verified that the internal

standard recovery percentages are equivalent to the surrogate recovery percentages.

Field Duplicate. A field duplicate is a second field sample collected from a selected monitoring well. Field

duplicate samples serve as a check on laboratory quality as well as potential variability of the sample matrix.

The field duplicate is analyzed and compared to the first sample to assess the precision of the analytical

method. This comparison can be expressed by the RPD between the original and duplicate sample.

Calibration. Satisfactory instrument calibration is established to confirm that an instrument is capable of

producing acceptable quantitative data. An initial calibration verification (ICV) demonstrates that the instrument is capable of acceptable performance at the beginning of an experimental sequence. Continuing

calibration verifies (CCV) that the daily performance of the instrument is satisfactory.

Internal Standard. An internal standard is a chemical substance that is added in a constant amount to

samples, the blank and calibration standards in a chemical analysis. This substance is then used for

calibration by plotting the ratio of the analyte signal to the internal standard signal as a function of the

analyte concentration of the standards. This is done to correct loss of analyte during sample preparation.

4.0 QA/QC Review Results

The following subsections document the results of the quality assurance review for each sampling event.

4.1 March 24, 2007 Event

The data reviewed includes storm water sample data collected during sampling performed on March 24,

2007. Samples were analyzed for one or more of the following: DOC, TOC, TSS, turbidity, metals, oil and

grease, phthalates, organochlorine pesticides, PAHs, PCB aroclors, and PCB congeners using the methods

listed in Section 2.0.

**Reporting Limits**. Elevated MRLs of analytes consisted of the following:

Organochlorine Pesticides: Some of the analyte MRLs were raised. The chromatogram indicated

the presence of non-target background components. The matrix interference prevented adequate

resolution of the target compounds at the MRL. The results were flagged with an "i".

Storm Water Data Summary Report Terminal 4 Slip 1 and Slip 3 Upland Facilities March 6, 2009 PCB Aroclors: Aroclors 1016, 1221, 1232, 1248, and 1254 for sample Basin R and Basin R Dissolved (Diss) were raised. The chromatogram indicated the presence of non-target background

components. The matrix interference prevented adequate resolution of the target compounds at

the MRL. The results were flagged with an"i".

MRLs were reviewed and are generally acceptable for this project. MRLs for individual samples varied

based on the magnitude of the chemical impact. The MRLs for some organochlorine pesticides and PAHs

were higher than the applicable screening level. These values are highlighted yellow.

Detection Limits and Estimated Concentrations. Several estimated MDLs and concentrations were

identified as follows:

Estimated non-detect concentrations, sample specific MDL, calculated for selected non-detected analytes:

PCB Congeners: Flagged with an asterisk.

Estimated detected concentrations below the lower calibration limit of the instrument:

PCB Congeners: Flagged with a "J" qualifier for samples Basin Q and Basin Q –Diss.

Estimated detected concentrations that are below the MRL and above the MDL:

Metals: Flagged with a "B" qualifier.

<u>Phthalates:</u> Flagged with a "J" qualifier.

Organochlorine Pesticides: Flagged with a "J" qualifier.

PAHs: Flagged with a "J" qualifier.

Holding Times. The hold time was exceeded for the turbidity analyses of the samples. The results are

flagged with a "J1" qualifier indicating that the detected values are estimation. The sampled storm event took place during the weekend, limiting the capability of lab submission during required hold times. Other

analyses were completed within specified hold times.

Custody Records and Sample Receipt. Some samples were received below required temperature of 4°C

and consistent with the accompanying COC.

Method Blanks. The results from the method blanks are summarized in the following table:

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Analysis	Analyte	Concentration
Phthalates	di-n-butyl phthalate	0.046 μg/L
Organochlorine Pesticides	alpha-BHC	0.082 ng/L
	hexachlorobenzene	0.39 ng/L
	beta-BHC	0.43 μg/L
	gamma-BHC (lindane)	0.0045 µg/L
PAHs	napthalene	0.0088 µg/L

The "J3" qualifier indicates an estimated value based upon Ash Creek QA/QC review. Concentrations were flagged with a "J3" if detected concentrations were less than or equal to five times the detected concentration from the method blanks.

**Laboratory Control Sample**. Percent recoveries of the LCS were within control limits for DOC, TOC, TSS, turbidity, metals, phthalates, oil and grease, PCB aroclors, PCB congeners and PAHs, except organochlorine pesticides. There was no LCS analyzed for metals.

Organochlorine Pesticdes: The recovery of hexachloroethane and hexachlorobutadiene were outside the control limits listed in the results summary. Limits are default values temporarily in use until sufficient data points are generated to calculate statistical control limits. Based on the method and historic data, the recoveries observed are in the range expected for this procedure. No further corrective action was taken.

Laboratory Control Sample Duplicate. Percent recoveries of the LCS were within control limits for oil and grease, PCB aroclors, and PAHs, except organochlorine pesticides. There was no LCSD analyzed for DOC, TOC, TSS, turbidity, phthalates, and PCB congeners.

Organochlorine Pesticides: The lower control criterion was exceeded for toxaphene, oxychlordane, cis-nonachlor, trans-nonachlor, and mirex. The results indicate a potential bias for results reported from this analytical batch. Reanalysis was not performed at the request of the PC. The data are flagged with an asterisk to indicate the discrepancy.

Matrix Spike Analyses. Recent recoveries of the MS/MSD were within control limits for metals, oil and grease, and phthalates, except metals. There was no MS/MSD analyzed for DOC, TOC, TSS, turbidity, organochlorine pesticides, PCB aroclors, and PCB congeners. In most cases this was due to insufficient sample volume to perform an MS/MSD. A LCS/LCSD was reported in lieu of the MS/MSD for these

samples.

• Metals: The control criterion for matrix spike recoveries of aluminum for sample Basin D is not

applicable. The analyte concentration was significantly higher than the added spike concentration,

preventing accurate evaluation of the spike recovery. No data are flagged.

Lab Duplicate. The lab duplicate for metals were within quality control limits, except silver. No lab duplicate for DOC, TOC, TSS, turbidity, phthalates, oil and grease, organochlorine pesticides, PAHs,

phthalates, PCB aroclors, and PCB congeners was analyzed.

The duplicate RPD criterion of 30% was exceeded for silver in the laboratory duplicate. The associated

results are flagged with a "J2" qualifier indicating that the analyte was positively identified and the

associated numerical value is the approximate concentration of the analyte in the sample.

**Surrogate Recovery.** Surrogate recoveries were within quality control limits.

Field Duplicate. The analytes which exceed an RPD of 30% were flagged with a "J2" qualifier. The

following are analytes and sample locations for precision goals exceedances:

Metals: Aluminum, arsenic, cadmium, chromium, lead, and nickel.

**Calibration**. Below is documentation of calibration outliers during the analysis of the samples:

• Organochlorine Pesticides: A dual column confirmation is used in the analysis for organochlorine

pesticides by 8081A, which produces two different results for each analysis. When the ICV criteria

are met for both columns, then the higher of the two samples results is generally reported.

The primary evaluation criteria were not met for chlordane, so the results were reported from the

column with the acceptable ICV. The case narrative reported that the data quality is not affected

and that no corrective action was necessary.

The primary evaluation criterion for the confirmation column was exceeded for tetrachloro-m-xylene

for the CCV. The results are reported from the column with an acceptable CCV.

The primary evaluation criteria were exceeded for a few analytes for their CCV. An alternative

evaluation was performed by using the average percent recoveral of analytes in the verification

standard. The standard meets the alternative evaluation criteria.

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Analytes in several samples were reported from a column using average percent recovery of the analytes in the verification standard.

At least one analyte in each sample was flagged with the "P" qualifier indicating that the instrument confirmation criteria were exceeded. The case narrative noted that the higher of the two values was reported when both peaks were within the expected retention time window for this analysis and Gaussian in shape or the lower of the two values was reported when there was an apparent interference on the alternate column that produced the higher value. A subset of samples were flagged with the qualifier "JP" indicating that the aforementioned confirmation comparison criteria are not applicable because at least one of the values is below the MRL.

 <u>PCB Aroclors:</u> The primary evaluation criterion was exceeded for aroclor 1016 in the continuing calibration verification. The alternative evaluation was performed using the average percent recovery of the analytes in the verification standard. The standard meets the alternative evaluation criteria.

The confirmation comparison criterion of 40% for dissolved aroclor 1242 was exceeded in sample Basin L. The lower of the two values was reported because of an apparent interference on the alternate column that produced the higher value. The associated result is flagged with the "P" qualifier.

**Internal Standard.** Below is documentation of the outliers:

• <u>Phthalates:</u> The internal standard recovery of chrysene-12, acenapthene-d10, and phenanthrene-d10 in sample Basin M was outside control criteria because of suspected matrix interference. The associated results are flagged with an asterisk.

**Other.** Case Narrative notes that apply to specific analysis or analytes.

PCB Congeners: The case narrative noted that when mixtures of PCB Aroclors are present in a sample, correct identification and quantitative analysis of the individual Aroclors can be subjective, in particular when differentiating between aroclor 1242 and 1248. The laboratory conducted a review of the sample chromatograms in a number of samples where 3 PCB Aroclors were detected and the chromatograms indicated the presence of PCB patterns that spanned the entire elution range from aroclor 1242 to aroclor 1260. The laboratory noted that when aroclor mixtures are present in a sample, care is taken to minimize the possibility of double-counting PCBs, but the potential exists for a high bias from contribution of one aroclor to another due to common peaks or peaks that cannot be completely resolved.

The case narrative noted that a number of results were flagged with an "I" qualifier noting chemical interference.

4.2 April 7, 2007 Event

The data reviewed includes storm water sample data collected during sampling performed on April 7, 2007.

Samples were analyzed for one or more of the following: DOC, TOC, TSS, turbidity, metals, oil and grease, phthalates, organochlorine pesticides, PAHs, PCB aroclors, and PCB congeners using the methods listed in

Section 2.0.

**Reporting Limits**. Elevated MRLs of analytes consisted of the following:

Organochlorine Pesticides: The chromatogram indicated the presence of non-target background

components. The matrix interference prevented adequate resolution of the target compounds at

the MRL. The results were flagged with an "i".

MRLs were reviewed and are generally acceptable for this project. MRLs for individual samples varied

based on the magnitude of the chemical impact. The MRLs for some organochlorine pesticides and one

PAH were higher than the applicable screening level. These values are highlighted yellow.

Detection Limits and Estimated Concentrations. Below is documentation of MDL and concentration

outliers during the analysis of the samples:

Estimated concentrations that are below the MRL and above the MDL:

Phthalates: Flagged with a "J" qualifier.

PAHs: Flagged with a "J" qualifier.

Holding Times. The hold time was exceeded for the turbidity analyses of the samples. The results are

flagged with a "J1" qualifier indicating that the detected values are estimation. The sampled storm event

took place during the weekend, limiting the capability of lab submission during required hold times. Other

analyses were completed within specified hold times.

Custody Records and Sample Receipt. The samples were received below required temperature of 4°C

and consistent with the accompanying COC.

**Method Blanks.** The results from the method blanks are summarized in the following table:

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Analysis	Analyte	Concentration
Phthalates	di-n-butyl phthalate	0.049 µg/L
	Benzyl butyl phthalate	0.015 µg/L
Organochlorine Pesticides	gamma-BHC (lindane)	0.41 µg/L
PCB-1	napthalene	28.2 pg/L

The "J3" qualifier indicates an estimated value based upon Ash Creek QA/QC review. Concentrations were flagged with a "J3" if detected concentrations were less than or equal to five times the detected concentration from the method blanks.

**Laboratory Control Sample.** Percent recoveries of the LCS were within control limits for TOC, TSS, turbidity, metals, phthalates, oil and grease, PCB aroclors, PCB congeners and PAHs, except organochlorine pesticides. There was no LCS analyzed for DOC. LCS performed for DOC is comparable to the LCS performed for TOC.

- Organochlorine Pesticides: The advisory criterion was exceeded for hexachloroethane and hexachlorbutadiene. These compounds were not included in the subset of analytes used to control the analysis. The recovery information reported for these analytes is for advisory purposes only. No further corrective action was required.
- The spike recovery of chlordane was outside the lower control criterion. This analyte was not including in the spiking mix. It was unknown at the time of extraction that this was a target analyte. Note that alpha-chlordane and gamma-chlordane, which are components of Chlordane, were spiked and yielded acceptable recoveries. The analyte in question was not detected in the associated field samples. Additional analyses of the associate field samples were not performed. The data are flagged to indicate the discrepancy with an asterik.

**Laboratory Control Sample Duplicate**. Percent recoveries of the LCS were within control limits for oil and grease, organochlorine pesticides, PCB aroclors and PAHs. There was no LCSD analyzed for DOC, TOC, TSS, turbidity, phthalates, and PCB congeners.

**Matrix Spike Analyses.** Percent recoveries of the MS/MSD were within control limits for DOC, TOC, oil and grease, and PAHs, except phthalates. There was no MS/MSD analyzed for TSS, turbidity, and PCB congeners. Due to insufficient volume, no field duplicate was analyzed for PCB Aroclors.

<u>Phthalates:</u> The matrix spike recovery of bis(2-ethylhexyl) phthalate in Basin D was outside control criteria. Recovery in the LCS was acceptable, which indicates the analytical batch was in control. The matrix spike outlier suggests a potential bias in this matrix. No further corrective action was appropriate.

- Organochlorine Pesticides: The control criteria for 2,4'-DDD, 2,4'DDE and 2,4'-DDT for sample
  Basin M Diss were not applicable. The chromatogram indicates non-target matrix background
  components are contributing to the reported matrix spike concentrations. Thus, the reported
  recoveries contain a high bias. Based on the magnitude of background contribution, the
  interference appears to be minimal.
- Organochlorine Pesticides: The matrix spike recoveries of several analytes for sample Basin Q
  Diss were outside control criteria because of matrix interference. The chromatogram indicated the
  presence of non-target background components that prevented adequate resolution of the target
  analytes. As a result, accurate quantitation was not possible. No further corrective action was
  required.

Lab Duplicate. The lab duplicates for DOC, TOC, TSS, and turbidity were within quality control limits. Due to insufficient volume, no lab duplicate was analyzed for phthalates. No lab duplicate was analyzed for oil and grease and PCB congeners.

The duplicate RPD criterion of 30% was exceeded for endrine aldehyde, aroclor 1254, and phenanthrene in the laboratory duplicates. The associated results are flagged with a "J2" qualifier indicating that the analyte was positively identified. The associated numerical value is the approximate concentration of the analyte in the sample.

In some samples, the RPD criterion of 30% is not applicable because the analyte concentration was not significantly greater than the MRL. Analytical values derived from measurements close to the MDL are not subject to the same accuracy and precision criteria as results derived from measurements higher on the calibration range for the method. The following is a list of analytes that fit this category:

- PCB Aroclors: Aroclor 1260;
- PAHs: Fluoranthene, pyrene, benz(a)anthracene, chrysene, benzo(b)fluoranthene, benzo(k)fluoranthene, benzo(a)pyrene, indeno(1,2,3-cd)pyrene, dibenzo(a,h)anthracene, benzo(g,h,i)perylene,

**Surrogate Recovery.** Surrogate recoveries were within quality control limits, except phthalates.

The surrogate recovery of triphenyl phosphate and perylene-d12 in sample Basin M and Basin R was outside control criteria because of suspected matrix interference. The sample contained particulate which has been shown to cause low recovery of the internal standard chrysened12. The low recovery of the internal elevates the calculated recovery of the surrogate. The results have been flagged to indicate the discrepancy.

**Field Duplicate.** The analytes which exceeded an RPD of 30% were flagged with a "J2" qualifier. The following are analytes and sample locations for precision goals exceedances:

- Organochlorine Pesticides: cis-nonachlor
- PCB Aroclors: Aroclors 1242, 1254, and 1260.

**Calibration.** Below is documentation of calibration outliers during the analysis of the samples:

 Organochlorine Pesticides: The primary evaluation criteria were not met on the confirmation column for chlordane. The ICV results are reported from the acceptable column. The data quality is not affected. No further corrective action was necessary.

The primary evaluation criterion was exceeded for the following analytes in CCV mirex, hexachloroethane, hexachlorobutadiene, and 4,4'-DDE. In accordance with CAS standard operating procedures, the alternative evaluation specified in the EPA method was performed using the average percent recovery of the analytes in the verification standard. The standard meets the alternative evaluation criteria.

Organochlorine Pesticides & PCB Aroclors: The confirmation comparison criterion of 40% difference for a few analytes was exceeded in the samples. The higher of the two values was reported when both peaks were within the expected retention time window for this analysis and Gaussian in shape or the lower of the two values was reported when there was an apparent interference on the alternate column that produced the higher value. The qualifier "JP" indicates the confirmation comparison criteria are not applicable because at least one of the values is below the MRL.

Internal Standard. The internal standard recovery of acenapthene-d10, chrysene-12, and phenanthrene-d10 in sample Basin R was outside control criteria because of suspected matrix interference. The internal standards are spiked pre-extraction and are subject to matrix interference. The sample contained particulate which has been shown to cause low recovery of certain compounds. Analytes associated with this internal standard may also have a biased result. The low internal standard recovery resulted in an elevated recovery of the surrogate triphenyl phosphate. The results quantified using this internal standard are flagged to indicate the discrepancy.

#### Other.

<u>PCB Aroclors:</u> The samples in this data set appear to have been subjected to environmental stresses such as weathering, causing a pattern degradation and changing the peak ratios. When pattern degradation occurs, correct identification and quantitative analysis of the individual aroclors can be subjective. Care was taken to report the aroclor with the best pattern match.

4.3 May 3, 2007 Event

The data reviewed includes storm water sample data collected during sampling performed on May 3, 2007.

Samples were analyzed for one or more of the following: DOC, TOC, TSS, turbidity, metals, oil and grease, phthalates, organochlorine pesticides, PAHs, PCB aroclors, and PCB congeners using the methods listed in

Section 2.0.

**Reporting Limits.** Elevated MRLs of analytes consisted of the following:

• Metals: A number of samples are elevated because the samples required dilution for analysis by

Inductively Coupled Plasma – Mass Spectroscopy (ICP-MS). Additionally, the MRL for selenium

for sample Basin R is elevated further due to suspected matrix interference.

• <u>Organochlorine Pesticides:</u> The MRL is elevated for the pesticide analytes in the May 3, 2007

samples. The sample extract was diluted prior to instrumental analysis due to relatively high levels

of non-target background components. Clean-up of the extract was performed within the scope of the method, but did not eliminate enough of the background components to prevent dilution. A

semiquantitative screen was performed prior to final analysis. The results of the screening

indicated the need to perform a dilution. The results are flagged with an "i" to indicate the anomaly.

The MRL is further elevated for a few pesticide analytes in several samples. The chromatogram

indicated the presence of non-target background components. The matrix interference prevented adequate resolution of the target compounds at the MRL. The results are flagged with an "i" to

indicate the anomaly.

• PCB Aroclors: The MRL is elevated for aroclor 1242 in samples Basin M Diss and Basin M-DUP

Diss. The chromatogram indicated the presence of non-target background components. The matrix interference prevented adequate resolution of the target compounds at the MRL. The

results are flagged to indicate matrix interference.

PAHs: The MRLs for numerous samples were elevated due to less than optimal sample volume

available for analysis.

MRLs were reviewed and are generally acceptable for this project. MRLs for individual samples varied

based on the magnitude of the chemical impact. The MRLs for some organochlorine pesticides and one

arsenic result were higher than the applicable screening level. These values are highlighted yellow.

**Holding Times**. Analyses were completed within specified hold times.

Detection Limits and Estimated Concentrations. Below is documentation of MDL outliers identified

during the QA/QC review:

Organochlorine Pesticides: The MDL is elevated for several analytes in the Method Blank. The chromatogram indicated the presence of non-target background components, which were apparently introduced as laboratory artifacts. The contamination prevented adequate resolution of the target compounds at the MDL. Note the level of background was relatively low compared to the MDL, so the affect on the results was minimal. The results are flagged with an "i" to indicate the anomaly.

**Custody Records and Sample Receipt.** The samples were received below required temperature of 4°C and consistent with the accompanying COC.

**Method Blanks**. The results from the method blanks are summarized in the following table:

Analysis	Analyte	Concentration
Metals	mercury	0.10 µg/L
	zinc	0.20 µg/L
Phthalates	di-n-butyl phthalate	0.082 µg/L
	Benzyl butyl phthalate	0.024 µg/L
	gamma-BHC (lindane)	4.5 ng/L
	heptachlor	5.5 ng/L
Organochlorine Pesticides	Endosulfan sulfate	13 ng/L
	2,4'-DDE	8.7 ng/L
	2,4'-DDT	3.4 ng/L
	napthalene	0.0070 ng/L
	2-methylnapthalene	0.0042 ng/L
	Acenaphtylene	0.0045 ng/L
	Acenaphthene	0.0052 ng/L
	Dibenzofuran	0.0059 ng/L
	Fluorine	0.0050 ng/L
PAHs	Phenanthrene	0.0073 ng/L
	Anthracene	0.0046 ng/L
	Fluoranthene	0.0073 ng/L
	Pyrene	0.0061 ng/L
	Benz(a)anthracene	0.0063 ng/L
	Benzo(a)pyrene	0.0043 ng/L
	Indeno(1,2,3-cd)pyrene	0.0062 ng/L
	Dibenz(a,h)anthracene	0.0055 ng/L
	Benzo(g,h,i)perylene	0.0060 ng/L

The "J3" qualifier indicates an estimated value based upon Ash Creek QA/QC review. Concentrations were flagged with a "J3" if detected concentrations were less than or equal to five times the detected concentration from the method blanks.

**Laboratory Control Sample.** Percent recoveries of the LCS were within control limits for TOC, TSS, turbidity, metals, phthalates, oil and grease, PCB aroclors, PCB congeners and PAHs, except organochlorine pesticides. There was no LCS analyzed for DOC. LCS performed for DOC is comparable to the LCS performed for TOC.

The spike recovery of chlordane was outside the lower control criterion. This analyte was not included in the spiking mix. It was unknown at the time of extraction that this was a target analyte. Note that alphachlordane and gamma-chlordane spikes, which are components of Chlordane, have acceptable recoveries. The analyte in question was not detected in the associated field samples. Additional analyses of the associate field samples were not performed at the request of the Project Chemist. The data is flagged with an asterisk to indicate the discrepancy.

Laboratory Control Sample Duplicate. Percent recoveries of the LCS were within control limits for oil and grease, PCB aroclors and PAHs, except organochlorine pesticides. There was no LCSD analyzed for DOC, TOC, TSS, turbidity, metals, phthalates, and PCB congeners.

 Organochlorine Pesticides: The control criterion was exceeded for hexachlorobenzene and 4,4'-DDD. The anomaly indicates a potential bias for results reported from this analytical batch. The following samples tested positive for analytes in question: Basin M, Basin M Diss, and Basin T Diss. Reanalysis was not performed because insufficient sample remained for additional testing. The data flagged with an asterisk to indicate the anomaly.

The RPD of 30% was exceeded for alpha-BHC, hexachlorobenzene, delta-BHC, heptachlor, 4,4'-DDE, and 4,4'-DDD. Reanalysis of the associated field samples could not be performed because insufficient sample remained for additional testing. The data are flagged with an asterisk to indicate the anomaly.

 PAHs: The RPD of 30% was exceeded for benzo(a)pyrene. The spike recoveries in the MS and LCS/LCSD were within acceptance limits, indicating the analytical batch was in control. No further corrective action was appropriate.

**Matrix Spike Analyses.** Percent recoveries of the MS/MSD were within control limits for TOC, metals, oil and grease, and phthalates, except organochlorine pesticides. There was no MS/MSD analyzed for DOC, TSS, turbidity, PAHs, and PCB congeners. Due to insufficient volume, no field duplicate was analyzed for PCB Aroclors.

- Organochlorine Pesticides: The control criteria for hexachlorobenzene, endosulfan I, alphachlordane, 4,4'-DDE, endrin aldehyde, and methxychlor for Batch QCMS indicate values outside control criteria.
- Organochlorine Pesticides: gamma-chlordane, dieldrin, endosulfan II, 4,4'-DDD, and 4,4'-DDT indicate the control criterion is not applicable.

**Lab Duplicate.** The lab duplicate for TOC, TSS, and turbidity were within quality control limits, except metals. No lab duplicate was analyzed for DOC, phthalates, oil and grease, organochlorine pesticides, PAHs, PCB aroclors, and PCB congeners.

The duplicate RPD criterion of 30% was exceeded for arsenic and zinc in the laboratory duplicate. The associated results are flagged with a "J2" qualifier indicating that the analyte was positively identified and the associated numerical value is the approximate concentration of the analyte in the sample.

**Surrogate Recovery.** Surrogate recoveries were within quality control limits, except for phthalates and PCB congeners.

 <u>Phthalates:</u> The upper control criterion was exceeded for 1,3-dimethyl 1,2-nitrobenzene in sample Basin T Diss. The elevated recovery may indicate a high bias. No analytes were detected above the MRL. No sample remained for re-extraction.

The lower control criterion was exceeded for perylene-d12 in sample Basin R Diss. The low recovery may indicate a low bias. No sample remained for re-extraction.

 <u>PCB Congeners:</u> The lower control criterion was exceeded for PCB-1 in the Method Blank. The low recovery may indicate a low bias. The surrogate recoveries and LCS were within control criteria for the other samples, thus establishing the validity of the data.

**Field Duplicate.** The analytes which exceeded an RPD of 30% were flagged with a "J2" qualifier. The following details the analyte and sample location for precision goals exceedances:

- Metals: aluminum, cadmium, and lead;
- Phthalates: benzyl butyl phthalate;
- PAHs: phenanthrene, indeno(1,2,3-cd)pyrene, dibenz(a,h)anthracene, and benzo(q,h,i)perylene;
- PCB Aroclors: 1242, 1254, and 1260;
- PCB Congeners Total: Detected PCBs between the sample pairs; and
- PCB Congeners Dissolved: PCB 43/49 and 106/118.

**Calibration.** Below is documentation of calibration outliers during the analysis of the samples.

- <u>Phthalate:</u> The upper control criterion was exceeded for di-n-octyl phthalate. The field samples analyzed in this sequence did not contain the analytes in question. Since the anomaly equates to a potential high bias, the data quality is not affected. Nor further corrective action was appropriate.
- Organochlorine Pesticides: The primary evaluation criterion was exceeded for the following analytes in CCV 0522 F016, 0522 F044, and 0522F063. In accordance with CAS standard operating procedures, the alternative evaluation specified in the EPA method was performed using the average percent recovery of the analytes in the verification standard. The standard meets the alternative evaluation criteria.

Results for decachlorobiphenyl, 2,4'-DDD, 2.4'-DDT have been reported from a column using average percent recovery of the analytes in the verification standard.

PCB Aroclors: The analysis of PCB aroclors by EPA 8082 requires the use of dual column confirmation. When the CCV criterion is met for both columns, the higher of the two sample results is generally reported. The primary evaluation criteria were not met on the confirmation column for decachlorobiphenyl in CCVs. The results are reported from the column with an acceptable CCV. The data quality is not affected. No further corrective action was necessary.

The primary evaluation criterion was exceeded for decachlorobiphenyl. In accordance with CAS standard operating procedures, the alternative evaluation specified in the EPA method was preformed using the average percent recovery of the analytes in the verification standard. The standard meets the alternative evaluation criteria.

Results for decachlorobiphenyl surrogate in the samples have been reported from a column using average percent recovery of analytes in the verification standard.

Internal Standard. The lower control criterion was exceeded for the internal standard chrysene-d12 in samples Basin R and Outfall 53. The lower control criterion was exceeded for phenanthrene d-10 in sample Basin R. The sample Outfall 53 was re-analyzed with acceptable internal standard recoveries. The internal standards are added pre-extraction and are subject to matrix interferences. The lower recovery is due typically to the particulate in the sample. The lower recovery of the internal standards may result in the elevated calculated recoveries of the target analytes associated with these internal standards. The analytes have been flagged to indicate the discrepancy.

### 4.4 May 20, 2007 Event

The data reviewed includes storm water sample data collected during sampling performed on May 20, 2007. Sample from Basin L was analyzed for one or more of the following: DOC, TOC, TSS, turbidity, metals, oil and grease, phthalates, organochlorine pesticides, PAHs, PCB aroclors, and PCB congeners using the

methods listed in Section 2.0. Other samples were analyzed for the general chemistry parameters (DOC, TOC, TSS, and turbidity)

TOC, TSS, and turbidity).

**Reporting Limits**. Elevated MRLs of analytes consisted of the following:

• Organochlorine Pesticides: The MRL is elevated for analytes in Batch QC K0704413-4MS/DMS.

The sample extract was diluted prior to instrumental analysis due to relatively high levels of non-

target background components. Clean-up of the extract was performed within the scope of the

method, but did not eliminate enough of the background components to prevent dilution. A

semiquantitative screen was performed prior to final analysis. The results of the screening

indicated the need to perform a dilution. The results are flagged with an asterisk to indicate the

matrix interference.

The MRL is further elevated for a few analytes in several samples. The chromatogram indicated

the presence of non-target background components. The matrix interference prevented adequate

resolution of the target compounds at the MRL. The results are flagged with an asterisk to indicate

matrix interference.

MRLs were reviewed and are generally acceptable for this project. MRLs for individual samples varied

based on the magnitude of the chemical impact. The MRLs for some organochlorine pesticides were higher

than the applicable screening level. These values are highlighted yellow.

Detection Limits and Estimated Concentrations. Below is documentation of MDL outliers identified

during the QA/QC review:

• Organochlorine Pesticides: The MDL is elevated for several analytes in the Method Blank. The

chromatogram indicated the presence of non-target background components, which were

apparently introduced as laboratory artifacts. The contamination prevented adequate resolution of

the target compounds at the MDL. Note the level of background was relatively low compared to

the MDL, so the effect on the results was minimal. The results are flagged with an asterisk to

indicate the anomaly.

**Holding Times**. Analyses were completed within specified hold times.

Custody Records and Sample Receipt. The samples were received below required temperature of 4°C

and consistent with the accompanying COC.

**Method Blanks**. The results from the method blanks are summarized in the following table:

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Analysis	Analyte	Concentration
Metals	chromium	0.07 μg/L
	zinc	0.14 µg/L
Phthalates	di-n-butyl phthalate	0.053 µg/L
	bis(2-ethylhexyl) phthalate	0.058 µg/L
Organoshlarina Dagticidas	hexachlorobenzene	0.34 ng/L
Organochlorine Pesticides	hexachloroethane	0.23 ng/L
PCB Aroclors	PCB-1	28.9 ng/L
L CD VIOCIOI2	PCB-77	3.74 ng/L

The "J3" qualifier indicates an estimated value based upon Ash Creek QA/QC review. Concentrations were flagged with a "J3" if detected concentrations were less than or equal to five times the detected concentration from the method blanks.

**Laboratory Control Sample.** Percent recoveries of the LCS were within control limits for TOC, TSS, turbidity, metals, phthalates, oil and grease, PCB congeners and PAHs, except organochlorine pesticides. There was no LCS analyzed for DOC and PCB aroclors. LCS performed for DOC is comparable to the LCS performed for TOC.

Organochlorine Pesticides: The control criterion was exceeded for 2,4'-DDE, 2,4'-DDD, and 2.4'-DDT. The anomaly indicates a potential bias for results from this analytical batch. The following samples tested positive for the analytes in question, Basin L. Reanalysis was not performed because insufficient sample remained for additional testing. The data are flagged to indicate the discrepancy.

**Laboratory Control Sample Duplicate.** Percent recoveries of the LCS were within control limits for TSS, oil and grease, and PAHs, except organochlorine pesticides. There was no LCSD analyzed for DOC, TOC, turbidity, metals, phthalates, PCB aroclors, and PCB congeners.

The RPD of 30% was exceeded for 2,4'-DDE and 2,4'-DDD. Reanalysis of the associated field samples could not be performed because insufficient sample remained for additional testing. The data are flagged to indicate the anomaly.

**Matrix Spike Analyses.** Percent recoveries of the MS/MSD were within control limits for the samples analyzed for TOC, oil and grease, and phthalates, except metals, organochlorine pesticides, and PCB aroclors. There was no MS/MSD analyzed for DOC, TSS, turbidity, PAHs, and phthalates.

• <u>Metals:</u> The control criteria for aluminum and zinc for sample Basin L are not applicable. The analyte concentration in the sample was significantly higher than the added spike concentration, preventing accurate evaluation of the spike recovery.

The matrix spike recovery of lead for sample Basin L was outside the CAS control criteria as a result of the particulate matter in the sample. The RPD for the replicate analysis supports this. Since the unspiked sample contained high analyte concentration relative to the amount spiked, the variability between replicates was sufficient to bias the percent recovery outside normal CAS control criteria. The associated QA/QC results indicate the analysis was in control. No further action was appropriate.

Organochlorine Pesticides: The matrix spike recoveries of heptachlor epoxide, 4,4'-DDE, and 4,4'-DDD were outside control criteria. Recovery in the LCS was acceptable, which indicates the analytical batch was within control parameters. The matrix spike outlier suggests a potential bias in the matrix. No further corrective action was appropriate.

The matrix spike recovery of toxaphene was outside control criteria because of matrix interference. The chromatogram indicated the presence of non-target background components that prevented adequate resolution of the target analytes. As a result, accurate quantitation was not possible. No further corrective action was required.

The RPD for heptachlor, 4,4'-DDE, and 4,4'-DDD was outside control criteria. The spike recoveries in the MS and LCS were within acceptance limits. No further corrective action was appropriate.

 <u>PCB Aroclors:</u> Aroclor 1016 indicates control criterion was not applicable. The results have been reported from a column using average percent recovery of the analytes in the verification standard.

**Lab Duplicate.** The lab duplicate for TOC, turbidity, and metals were within quality control limits. No lab duplicates were analyzed for DOC, TSS, phthalates, oil and grease, organochlorine pesticides, PAHs, and PCB aroclors.

**Surrogate Recovery.** Surrogate recoveries were within quality control limits, except for organochlorine pesticides.

The control criteria for Batch QC K0704413-4 MS/DMS and the corresponding surrogates decachlorobiphenyl and tetrachloro-m-xylene, are not applicable. The analysis of the sample required dilution, which resulted in a surrogate concentration below the MRL. No further corrective action was appropriate.

**Field Duplicate**. Due to insufficient volume, no field duplicate was analyzed.

**Calibration**. Below is documentation of calibration outliers during the analysis of the samples.



Organochlorine Pesticides:

The primary evaluation criterion was exceeded for toxaphene, decachlorobiphenyl,

hexachlorobenzene, tetrachloro-m-xylene, and 4,4'-DDE. In accordance with CAS standard operating procedures, the alternative evaluation was preformed using the average percent

recovery of the analytes in the verification standard. The standard meets the alternative evaluation

criteria.

Results for toxaphene in the LCS and MS/MSD have been reported from a column using average

percent recovery of the analytes in the verification standard.

PCB Aroclors:

The primary evaluation criterion was exceeded for aroclor 1016. In accordance with CAS standard

operating procedures, the alternative evaluation specified in the EPA method was preformed using

the average percent recovery of the analytes in the verification standard. The standard meets the

alternative evaluation criteria.

**Internal Standard**. The lower control criterion was exceeded for the internal standards acenaphthene-d10,

phenanthrene-d10, and chrysene-d12 in samples Basin L. The low recoveries are a result of matrix interference and particulate during the extraction process. The sample contained heavy particulate.

The extraction disk plugged during the extraction process. Approximately 50 mls of sample was lost during

the extraction process. Only 5 mls of solvent was recovered from the elution step of the disk. The internal

standards are spike pre-extraction and are exposed to the matrix issues of the sample.

Other.

• Organochlorine Pesticides: The confirmation criterion of 40% difference for a few analytes was

exceeded in most samples. The higher of the two values was reported when both peaks were

within the expected retention time window for this analysis and Gaussian in shape or the lower of

the two values was reported when there was an apparent interference on the alternate column that

produced the higher value. The qualifier " ${\sf JP}$ " indicates the confirmation comparison criterion is not

applicable because at least one of the values is below the MRL.

PCB Aroclors: The confirmation comparison criterion of 40% difference for aroclor 1242 was

exceeded in sample Basin L Diss. The higher of the two values is reported because no evidence

of a matrix interference was observed.

4.5 September 28, 2007 Event

The data reviewed includes storm water sample data collected during sampling performed on September 28, 2007. Samples were analyzed for one or more of the following: DOC, TOC, TSS, turbidity, metals, oil

and grease, phthalates, organochlorine pesticides, PAHs, PCB aroclors, and PCB congeners using the

methods listed in Section 2.0.

Reporting Limits. Elevated MRLs of analytes consisted of the following:

• Organochlorine Pesticides: The chromatogram indicated the presence of non-target background

components. The matrix interference prevented adequate resolution of the target compounds at

the MRL. The results were flagged with an "i".

<u>PCS Aroclors:</u> PCB aroclors were raised for the samples during this event. The chromatogram

indicated the presence of non-target background components. The matrix interference prevented

adequate resolution of the target compounds at the MRL. The results were flagged with an "i".

MRLs were reviewed and are generally acceptable for this project. MRLs for individual samples varied

based on the magnitude of the chemical impact. The MRLs for some organochlorine pesticides and one

metal were higher than the applicable screening level. These values are highlighted yellow.

**Detection Limits and Estimated Concentrations.** No factors affecting the MDL have been identified.

**Holding Times**. Analyses were completed within specified hold times.

Custody Records and Sample Receipt. The samples were received below required temperature of 4°C

and consistent with the accompanying COC.

**Method Blanks**. The results from the method blanks are summarized in the following table:

Analysis	Analyte	Concentration
Metals	Aluminum	1.1 µg/L
	Cadmium	0.010 µg/L
	Chromium	0.09 µg/L
	Lead	0.02 µg/L
	Zinc	0.32 µg/L
	di-n-octyl phthalate	0.021 µg/L
Phthalates	Diethyl phthalate	0.021 µg/L
	di-n-butyl phthalate	0.071 μg/L
	Benzyl butyl phthalate	0.029 µg/L
Organochlorine Pesticides	4,4'-DDT	3.1 ng/L
	2,4'-DDT	0.75 ng/L
PAHs	naphthalene	0.0043 µg/L
	benz(a)anthracene	0.0045 µg/L
	benzo(b)fluoranthene	0.0039 µg/L
	benzo(k)fluoranthene	0.0026 µg/L
	indeno(1,2,3-cd)pyrene	0.0034 µg/L
	benzo(g,h,i)perylene	0.0036 µg/L

The "J3" qualifier indicates an estimated value based upon Ash Creek QA/QC review. Concentrations were flagged with a "J3" if detected concentrations were less than or equal to five times the detected concentration from the method blanks.

**Laboratory Control Sample.** Percent recoveries of the LCS were within control limits for TOC, TSS, turbidity, metals, phthalates, oil and grease, organochlorine pesticides, PCB aroclors, PCB congeners, and PAHs. There was no LCS analyzed for DOC and metals. LCS performed for DOC is comparable to the LCS performed for TOC.

**Laboratory Control Sample Duplicate**. Percent recoveries of the LCS were within control limits for TSS, oil and grease, organochlorine pesticides, PCB aroclors, and PAHs. There was no LCSD analyzed for TOC, turbidity, metals, phthalates, and PCB congeners.

**Matrix Spike Analyses.** Percent recoveries of the MS/MSD were within control limits for DOC, TOC, phthalates, PCB aroclors and PAHs, except metals. There was no MS/MSD analyzed for TSS, turbidity, oil and grease, organochlorine pesticides, and PCB congeners.

• <u>Metals:</u> The control criteria for matrix spike recoveries of aluminum for sample Basin M are not applicable. The analyte concentration was significantly higher than the added spike concentration,

preventing accurate evaluation of the spike recovery. No further corrective action was necessary. No data were flagged.

Lab Duplicate. The lab duplicate for TOC, turbidity, and metals were within quality control limits. No lab duplicate was analyzed for DOC, TSS, phthalates, oil and grease, organochlorine pesticides, PAHs, PCB aroclors, and PCB congeners.

**Surrogate Recovery.** Surrogate recoveries were within quality control limits with the exception of PCB congeners.

<u>PCB Congeners:</u> The lower control criterion was exceeded for PCB-155 in the Method Blank. The
low recovery may indicate a low bias. The surrogate recoveries and LCS were within control
criteria for the other samples, thus establishing the validity of the data.

Field Duplicate. Due to insufficient volume, no field duplicate was analyzed.

**Calibration Verification.** Below is documentation of calibration outliers during the analysis of the samples.

 Organochlorine Pesticides: The analysis of chlorinated pesticides by EPA 8081 requires the use of dual column confirmation. When the CCV criterion is met for both columns, the higher of the two sample results is generally reported.

The primary evaluation criteria were not met on the confirmation column for decochlorobiphenyl. The results are reported from the column with an acceptable CCV. The data quality is not affected. No further corrective action was necessary.

The primary evaluation criteria were exceeded for decachlorobiphenyl, tetrachloro-m-xylene, alpha-BHC, gamma-BHC, and methoxychlor in two of the CCV procedures. The alternative evaluation was performed using the average percent recoveral of the analytes in the verification standard. The standard meets the alternative evaluation criteria.

At least one analyte in the samples were flagged with the "P" qualifier indicating that the GC or HPLC confirmation criteria were exceeded. The higher of the two values is reported because no evidence of matrix interference was observed or the lower of the two values was reported if an apparent interference on the alternate column produced the higher value.

A number of analyte results from samples Basin Q and Basin Q-dissolved were flagged with the qualifier "JP" indicating that the aforementioned confirmation comparison criteria are not applicable because at least one of the values is below the MRL.

Decachlorbiphenyl, tetrachloro-m-xylene, alpha-BHC, gamma-BHC, and methoxyclor in samples Basin L, Basin M, Basin Q, and Basin Q-Diss have been reported from a column using average percent recovery of the analytes in the verification standard.

• <u>PCB Aroclors:</u> The primary evaluation criterion was exceeded for aroclor 1260 in the CCV. The alternative evaluation was performed using the average percent recovery of the analytes in the

verification standard. The standard meets the alternative evaluation criteria.

Internal Standard. Internal standards were within quality control limits.

4.6 November 16, 2007 Event

The data reviewed include storm water sample data collected during sampling performed on November 16,

2007. Samples were analyzed for one or more of the following: DOC, TOC, TSS, turbidity, metals, oil and

grease, phthalates, organochlorine pesticides, PAHs, PCB aroclors, and PCB congeners using the methods

listed in Section 2.0.

**Reporting Limits**. Elevated MRLs due to matrix interference consisted of the following:

• Organochlorine Pesticides for Basin T: toxaphene, 2,4'-DDD, heptachlor and aldrin;

• Organochlorine Pesticides for Basin T DUP: delta-BHC, toxaphene, 2,4'-DDD, heptachlor epoxide,

endosulfan sulfate, methoxychlor, oxychlordane and aldrin; and

• PCB Aroclors for the Method Blank: aroclors 1221.

MRLs were reviewed and are generally acceptable for this project. MRLs for individual samples varied

based on the magnitude of the chemical impact. The MRLs for some organochlorine pesticides and PAHs

were higher than the applicable screening level. These values are highlighted yellow.

**Detection Limits and Estimated Concentrations.** No factors affecting the MDL have been identified.

Holding Times. The samples were analyzed within the holding times specified for each analyses

performed.

Custody Records and Sample Receipt. The samples were received below required temperature of 4°C

and consistent with the accompanying COC.

Method Blanks. The results from the method blanks are summarized in the following table:

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Analysis	Analyte	Concentration
	Arsenic	0.01 μg/L
Metals	Cadmium	0.008 µg/L
	Zinc	0.3 μg/L
Phthalates	di-n-butyl phthalate	0.18 μg/L
Fillialates	Benzyl butyl phthalate	0.018 µg/L
	4,4'-DDE	3.1 ng/L
Organochlorine Pesticides	4,4'-DDT	Identified in Case Narrative, missing from analysis packet.
	2,4'-DDT	Identified in Case Narrative, missing from analysis packet.
DCD Congoners	PCB-81	2.15 pg/L
PCB Congeners	PCB-189	4.13 pg/L

The "J3" qualifier indicates an estimated value based upon Ash Creek QA/QC review. Concentrations were flagged with a "J3" if detected concentrations were less than or equal to five times the detected concentration from the method blanks.

**Laboratory Control Sample.** Percent recoveries of the LCS were within control limits for TOC, TSS, turbidity, phthalates, oil and grease, organochlorine pesticides, PCB congeners and PAHs. There was no LCS analyzed for DOC, metals, and PCB aroclors. LCS performed for DOC is comparable to the LCS performed for TOC.

**Laboratory Control Sample Duplicate**. Percent recoveries of the LCS were within control limits for phthalates, oil and grease, organochlorine pesticides, and PAHs. There was no LCSD analyzed for TOC, TSS, turbidity, metals, PCB aroclors, and PCB congeners.

**Matrix Spike Analyses.** Percent recoveries of the MS/MSD were within control limits for TOC and oil and grease, except PAHs and metals. There was no MS/MSD analyzed for TSS, turbidity, phthalates, PCB aroclors, and PCB congeners.

- <u>PAHs:</u> The control criterion for matrix spike recovery of acenaphthene is not applicable. The analyte concentration in the sample was significantly higher than the added spike concentration, preventing accurate evaluation of the spike recovery. No further corrective action was appropriate.
- Metals: The control criteria for matrix spike recoveries of aluminum and zinc for sample Basin R
  DUP are not applicable. The analyte concentration was significantly higher than the added spike
  concentration, preventing accurate evaluation of the spike recovery. No further corrective action
  was appropriate.

**Lab Duplicate.** The lab duplicate for TOC, TSS, and turbidity were within quality control limits, except metals. No lab duplicate was made for phthalates, organochlorine pesticides, PAHs, PCB aroclors, and PCB congeners.

A lab duplicate was made from the sample collected from Basin R DUP for metal analyses, in which silver exceeded the quality control limit of 30%. The analytes which exceeded an RPD of 30% were flagged with a "J2" qualifier.

**Surrogate Recovery.** Surrogate recoveries were within quality control limits for phthalates. The control criteria for the decachlorobiphenyl surrogate during analysis for PCB aroclors from sample Basin T Dup and two method blanks were outside quality control limits. The chromatogram indicated the presence of target/non-target background components that masked the surrogate, which prevented adequate resolution of quantification. Since the anomaly equates to a high bias, the data quality is not significantly affected. No further corrective action was appropriate.

**Field Duplicate.** The analytes which exceeded an RPD of 30% were flagged with a "J2" qualifier. The following details the analyte and sample location for precision goals exceedances:

- Metals from Basin R: Aluminum, chromium, and lead;
- <u>Phthalates from Basin D:</u> Bis(2-ethylethyene)phthalate;
- Oil and Grease from Basin D; and
- PAHs from Basin R: Fluoranthene, pyrene, chrysene, and benzo(b)fluoranthene.

**Calibration.** During analysis for organochlorine pesticides, the primary evaluation criterion was exceeded for several analytes. The alternative evaluation was performed using the average percent recovery of the analytes in the verification standard. The standard meets the alternative evaluation criteria. The data were flagged with an asterisk.

### 4.7 January 16, 2008 Event

The data reviewed includes storm water sample data collected during sampling performed on January 16, 2008. Sample from Basin D was analyzed for PCB congeners using the method listed in Section 2.0.

**Reporting Limits.** In some cases, the PCB results were flagged with an asterisk or "J" to indicate a sample-specific MRL in lieu of the MRL or detection below the low calibration limit, respectively. Other congeners were reported to the MRL, which corresponds to the low-point in the initial calibration curve.

MRLs were reviewed and are generally acceptable for this project. MRLs for individual samples varied based on the magnitude of the chemical impact. It is not expected that any of the elevated MRLs

compromised the usability of the data.

**Detection Limits and Estimated Concentrations.** No factors affecting the MDL have been identified.

Holding Times. The samples were analyzed within the holding times specified for the PCB congener

analysis.

Custody Records and Sample Receipt. The samples were received below required temperature of 4°C

and consistent with the accompanying COC.

Method Blanks. The results from the method blank detected PCB-189, tetraPCB, and total PCB at a

concentration of 2.76 pg/L. If detected concentrations were less than or equal to five times the detected concentration from the method blank (2.76 pg/L), the result was flagged with a "J3" qualifier. This qualifier

indicates an estimated value based upon Ash Creek QA/QC review.

Laboratory Control Sample. Percent recoveries of the LCS were within control limits.

Laboratory Control Sample Duplicate. There was no LCSD analyzed.

Matrix Spike Analyses. There was no MS/MSD analyzed.

**Lab Duplicate**. There was no lab duplicate analyzed.

**Surrogate Recovery.** Surrogate recoveries were within quality control limits.

**Field Duplicate**. Due to insufficient volume, no field duplicate was analyzed.

**Calibration**. Calibration standards were within quality control limits.

4.8 January 25, 2008 Event

The data reviewed includes storm water sample data collected during sampling performed on January 25,

2008. Sample from Basin D was analyzed for PCB congeners using the method listed in Section 2.0.

Reporting Limits. In some cases, the PCB results were flagged with an asterisk or "J" to indicate a

sample-specific detection limit (DL) in lieu of the MRL. Other congeners were reported to the MRL, which

corresponds to the low-point in the initial calibration curve.

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MRLs were reviewed and are generally acceptable for this project. MRLs for individual samples varied

based on the magnitude of the chemical impact. It is not expected that any of the elevated MRLs

compromised the usability of the data.

**Detection Limits and Estimated Concentrations.** No factors affecting the MDL have been identified.

Holding Times. The samples were analyzed within the holding times specified for the PCB congener

analysis.

Custody Records and Sample Receipt. The samples were received below required temperature of 4°C

and consistent with the accompanying COC.

Method Blanks. The results from the method blank detected PCB-77, tetraPCB and total PCBs at a

concentration of 6.24 pg/L. If detected concentrations were less than or equal to five times the detected concentration from the method blank (6.24 pg/L), the result was flagged with a "J3" qualifier. This qualifier

indicates an estimated value based upon Ash Creek QA/QC review.

**Laboratory Control Sample.** Percent recoveries of the LCS were within control limits.

**Laboratory Control Sample Duplicate**. There was no LCSD analyzed.

Matrix Spike Analyses. There was no MS/MSD analyzed.

**Lab Duplicate**. There was no lab duplicate analyzed.

Surrogate Recovery. Surrogate recoveries were within quality control limits with the exception of PCB-1 in

the sample Basin D Filtered. The low recovery may indicate a low bias. The remaining surrogate

recoveries for the sample and the batch LCS were within control criteria, thus establishing the validity of the

data.

**Field Duplicate.** Due to insufficient volume, no field duplicate was analyzed.

**Calibration**. Calibration standards were within quality control limits.

## 5.0 Qualifiers

Below is a list of all qualifiers used on the tabulated results of the stormwater analyses.

#### 5.1 Universal Qualifiers

- # = The control criteria is not applicable. See Case Narrative.
- I = The MRL/MDL has been elevated due to matrix or chromatogram interference.
- J1 = Hold time was exceeded for this analysis, the resulting value is estimated.
- J2 = The analyte was positively identified; the resulting concentration is an estimated value. The RPD exceeded the precision goal from the field or lab duplicate.
- J3 = The detected concentration of this analyte is equal to or less than 5 times the concentration detected in the Method Blank.
- J4 = The detected concentration of this analyte is equal to or less than 5 times the concentration detected in the Filter Blank.
- J5 = More than one of the three surrogate recoveries was outside control criteria, likely due to matrix interference.
- J6 = The Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD) recovery for this analyte exceeded the control criteria.
- J7 = The Matrix Spike/Matrix Spike Duplicate (MS/MSD) recovery for this analyte exceeded the control criteria.
- U = The compound was analyzed for, but was not detected ("non-detect") at or above the MRL/MDL.
- X = See Case Narrative.

#### 5.2 Inorganic Qualifiers

- \* = The result is an outlier. See Case Narrative.
- B = The analyte was found in the associated method blank at a level that is significant relative to the sample result.
- E = The result is an estimated amount because the value exceeded the instrument calibration range.
- J = The result is an estimated concentration that is less than the MRL but greater than or equal to the MDL.

#### 5.3 Organic Qualifiers

- \* = The result is an outlier. See Case Narrative.
- D = The reported result is from a dilution.
- P = The GC or HPLC confirmation criteria was exceeded. The relative percent difference is greater than 40% between the two analytical results.

#### 5.4 Metals Qualifiers

- B = The result is an estimated concentration that is less than the MRL but greater than or equal to the MDL.
- N = The matrix spike sample recovery is not within control limits. See Case Narrative.

### 5.5 PCB Congener Qualifiers

- B = This compound was also detected in the method blank.
- D = The amount reported is the maximum possible concentration due to possible chlorinated diphenylether interference.
- E = The reported value exceeds the calibration range of the instrument.
- H = The signal to noise ratio is greater than 10:1.
- I = Chemical interference.
- J = The amount detected is below the Lower Calibration Limit of the instrument.
- \* = Sample-specific detection limit (DL) in the MRL column. DLs were calculated for non-detected congeners.

#### **Abbreviations:**

CAS = Columbia Analytical Services

CCV = Continuing calibration verification

COC = Chain of custody

DEQ = Department of Environmental Quality

DL = Sample-specific estimated detection limit

DOC = Dissolved organic carbon

EPA = Environmental Protection Agency

ICP-MS = Inductively Coupled Plasma - Mass Spectroscopy

ICV = Initial calibration verification

LCS = Lab control sample

LCSD = Lab control sample duplicate

MDL = Method detection limit

μg/L = micrograms per liter

MRL = Method reporting limit

MS = Matrix spike

MSD = Matrix spike duplicate

ng/L = nanograms per liter

PAH = polynuclear aromatic hydrocarbon

PCB = Polychlorinated biphenyl

pg/L = picograms per liter

QA/QC = Quality assurance/Quality check

RPD = Relative percent difference

TOC = Total organic carbon

TPH = Total petroleum hydrocarbon

TSS = Total suspended solids